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समुद्री नमकीन से  
हाइड्रेटेड कैल्शियम सल्फेट — विशिष्टि  
( पहला पुनरीक्षण )

Hydrated Calcium Sulphate From  
Marine Brine — Specification  
( First Revision )

ICS 71.060

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## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Hydrated calcium sulphate from marine brine known in the trade as 'marine gypsum' is lifted from salt works and it is rain-washed to reduce the sodium chloride content and other soluble impurities. At present it is used by cement industries in place of mineral gypsum.

This Standard was originally published in 1967. This Standard is revised to update the reference clause, packing and marking clauses. Further, amendment has been incorporated and the instrumental test method for chloride determination has been included.

The composition of the committee responsible for formulation of this standard is given at Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## HYDRATED CALCIUM SULPHATE FROM MARINE BRINE — SPECIFICATION

( First Revision )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for hydrated calcium sulphate from marine brine.

### 2 REFERENCES

The standards listed below contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below:

<i>IS No.</i>	<i>Title</i>
1288 : 1982	Methods of test for mineral gypsum ( <i>second revision</i> )
1289 : 1960	Methods for sampling of mineral gypsum
1290 : 1973	Specification for mineral gypsum ( <i>second revision</i> )

### 3 REQUIREMENTS

**3.1 Description** — The material shall consist essentially of calcium sulphate dihydrate obtained from marine salt works.

**3.2** The material when dried as prescribed in **4** of IS 1288 shall comply with the requirements laid down in Table 1, when tested according to the methods prescribed in IS 1288.

### 4 PACKING AND MARKING

#### 4.1 Packaging

The material shall be packed in closed containers as agreed to between the purchaser and the supplier.

#### 4.2 Marking

**4.2.1** The packages shall be securely closed and bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade mark, if any;
- c) Gross and net mass;
- d) Date of manufacture; and
- e) Batch number.

#### 4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

**Table 1 Requirements for Hydrated Calcium Sulphate**

(Clause 3.2, 5.3.2, 5.3.11, and Annex A)

SI No. (1)	Characteristics (2)	Requirement (3)	Method of Test, Ref. to (4)
i)	Calcium sulphate, dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), percent by weight, <i>Min</i>	85	Clause <b>12</b> of IS 1288
ii)	Chlorides (as NaCl), percent by weight, <i>Max</i>	1.0	Annex A or clause <b>14</b> of IS 1288
iii)	Filtrability, seconds, <i>Max</i> (when specifically required by the purchaser)	50	Clause <b>15</b> of IS 1288

NOTE — A limit of 0.5 percent, *Max*, has been prescribed in IS 1290 for Grade 4 of mineral gypsum suitable for cement industry. This does not mean that the quality of marine gypsum specified in this standard is inferior to that specified in IS 1290 for use in cement industry.

## 5 SAMPLING

### 5.1 Sampling Procedure

Representative samples of the material shall be drawn as prescribed in IS 1289.

### 5.2 Number of Tests

The number of tests shall be in accordance with 6 of IS 1289.

### 5.3 Criteria for Conformity

#### 5.3.1 For Individual Samples

For calcium sulphate tested on the individual samples, the mean ( $\bar{X}$ ) and range ( $R$ ) of test results shall be

computed (the mean being the average of all and the range being the difference between the maximum and the minimum of the test results).

**5.3.1.1** The lot shall be declared as conforming to the requirement if the value of the expression ( $\bar{X} - 0.6 R$ ) is not less than the minimum value specified in Table 1.

#### 5.3.2 For Composite Sample

For chlorides tested on the composite sample, the lot shall be declared as conforming to the requirements for chlorides if the test result does not exceed the maximum value specified in Table 1.

## ANNEX A

(Clause 3.2, Table 1)

## DETERMINATION OF CHLORIDES

**A-1 ION CHROMATOGRAPHY FOR CHLORIDES****A-1.1 Principle**

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

**A-1.2 Equipments**

**A-1.2.1 Anion Guard Column** — A protector of the separator column.

**A-1.2.2 Anion Separator Column** — Suitable for selective separation of ions under analysis.

**A-1.2.3 Anion Suppressor Device** — Anion micromembrane suppressor is used to analyse the data.

**A-1.2.4 Detector** — Conductivity detector.

**A-1.2.5 Software** — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

**A-1.2.6 Sample loop** of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

**A-1.3 Reagents**

**A-1.3.1** Glass or polyethylene sample bottles.

**A-1.3.2** Distilled water or deionized water free from the anions of interest.

**A-1.3.3 Eluent** — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 l of water.

**A-1.3.4 Micromembrane Suppressor Solution** — (0.025 N of sulphuric acid) - Dilute 2.8 ml of conc. sulphuric acid in 4 l of water.

**A-1.4 Standard Solutions**

Chloride — Dissolve NaCl, 1.648 5 g in 1 l of reagent water.

**A-1.5 Calibration and Standardization**

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 - 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

**A-1.6 Procedure**

Dissolve between 1 to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 - 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used:

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no

deviation from the method.

**A-1.7 Data Analysis and Calculations**

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and

compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

**ANNEX B***(Foreword)***COMMITTEE COMPOSITION**

Inorganic Chemicals Sectional Committee, CHD 01

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*Member Secretary*

SAGAR SINGH  
SCIENTIST 'C' (CHD), BIS





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### Amendments Issued Since Publication

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